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## PATENT APPLICATION

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the application of:

Attorney Docket No.: 2950.25US01

Kumar et al.

Confirmation No.: 1810

Application No.: 09/136,483

Examiner: Michael A. Marcheschi

Filed: August 19, 1998

Group Art Unit: 1755

For: ALUMINUM OXIDE PARTICLES

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DECLARATION UNDER 37 C.F.R. § 1.132

Mail Stop RCE  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

I, Weidong Li, Ph.D., hereby declare as follows:

1. I am presently employed as a Senior Process Engineer at NanoGram Corporation and have worked at NanoGram since 2005.
2. I have a Ph.D. from University of Delaware in Material Science and have further experience as a Postdoctoral Research Scientist at the Center for Composite Materials, in Newark Delaware.
3. I have also previously worked in the area of nanotechnology while obtaining a Masters Degree at the Institute of Metal Research, Chinese Academy of Sciences, Shenyang, China. I have been coauthor on 11 refereed journal articles and two book chapters. A copy of my resume is attached.

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4. I have reviewed pending U.S. Patent application serial number 09//136,483 ('483 application). I am not an inventor on this patent application, and I did not participate in any of the work associated with this patent application.
5. I have conducted a literature search related to submicron scale aluminum oxide. As a result of this search, I found two relevant articles before 1999. I understand that these articles will be submitted to the patent office for consideration in this application. It is my conclusion that neither of these references disclose or suggest the materials presently claimed in the '483 application. Specifically, the Borsella article teaches a laser pyrolysis approach with precursors that result in amorphous particles and a carbon coating. These particles required heating at high temperatures to obtain crystalline  $\text{Al}_2\text{O}_3$ . The particles had a broad particle size distribution as shown in Fig. 3, so that these particles clearly are distinct from the claimed particles of the '483 application. The Kumar et al. article describes an arc plasma synthesis approach. As noted in the abstract, their particles are a core of metallic aluminum surrounded by a shell of  $\text{Al}_2\text{O}_3$ . As can be seen in Figs. 2(a) and 2(c), the particle size distribution is considerable broader than Applicants' claimed distributions. The micrograph shown in Fig. 2(a) strongly suggests larger particles that are precluded from this particular view.
6. I discussed the availability of Nanoparticles of aluminum oxide with Professor Pratsinis of the ETH/Swiss Federal Institute of Technology, Zurich, Switzerland. Professor Pratsinis is a well known expert on the vapor and liquid synthesis of nanoparticles. Professor Pratsinis pointed me to potential sources to explore for these materials.
7. I researched further the availability of commercial aluminum oxide powders. I found that Degussa started selling nanoscale aluminum oxide this year. Upon further research, I found that Aldrich Chemical sold nanoscale aluminum oxide. I could not determine what year these materials were first sold. However, I obtained a sample of these materials. Two scanning electron micrographs of these materials are shown. Some of the clearer particles are labeled with

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respect to size. However, as seen throughout the micrograph, there are a large number of smaller particles, and at least some of the larger particles seem to be fused agglomerates of small crystallites indicating that there is a large degree of fusing of the particles. Even the most optimistic evaluation of these particles indicates that the distributions are very significantly broader than the distributions claimed in the '483 application. Therefore, even particles obtainable more than seven years later than the filing date of the '483 patent cannot meet the properties of the materials claimed in the '483 patent.

8. I concluded from all of my research that the materials claimed in the '483 patent were not publicly available from any known source at the filing date of the '483 patent.

9. I declare that all statements made herein that are of my own knowledge are true and that all statements that are made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,



Weidong Li, Ph.D.


## CERTIFICATE OF FACSIMILE TRANSMISSION

I hereby certify that this paper is being transmitted by facsimile to the U.S. Patent and Trademark Office, Fax No. 571-273-6300 on the date shown below thereby constituting filing of same.

Date

December 21, 2006

Peter S. Dardi



Weidong Li's C.V. (Email: [weidong2004@gmail.com](mailto:weidong2004@gmail.com))

## WEIDONG LI (Ph.D.)

### CONTACT INFORMATION (1)

1788 Swanston Way  
San Jose, CA 95132  
302-897-5578 (mobile)  
[wqli@nanogram.com](mailto:wqli@nanogram.com)

### SUMMARY OF QUALIFICATION:

Have more than twelve-year research experience in the field of synthesis, characterization, and application of nanomaterials and thin films.

Expertise in:

- Synthesis techniques: laser pyrolysis (LP), chemical vapor deposition (CVD), physical vapor deposition (PVD), sol-gel, electrospinning, and rapid solidification (RSD).
- Characterization: XPS, XRD, TEM/SEM/EDS, AFM, DLS, FTIR, UV-VIS, DSC, and HPLC.
- Materials design: Structure-property relationships for a wide range of materials such as electronic, optical, magnetic, and catalytic materials.
- Surface science, nanoscience, vacuum science, thin films, solid state physics, polymer physics, physical chemistry, photochemistry, photocatalysis, phosphors, magnetism, device principles, optoelectronics, and electronic materials processing, etc.

### EXPERIENCE:

#### Senior Process Engineer (2005-Present)

NANOGRAM CORPORATION, San Jose, CA, 95134

1. Process engineer for advanced nanomaterials synthesis and characterization: Process development and optimization of nanoscale materials synthesis using the NanoGram's proprietary process, development and optimization of technology to treat these nanoscale materials to enable their use in product applications, scheduling development activities, managing technicians who operate advanced materials synthesis equipment and also communicating weekly progress internally and to the customer in meetings.

#### Postdoctoral Fellow (2004 – 2005)

CENTER FOR COMPOSITE MATERIALS, Newark, DE, 19716

1. Electrospinning of nanofibers and application of nanofibers in composite materials.
2. Study of surface sizing of glass fibers by nanoparticles with silane for impact resistance enhancement of composites
3. Surface characterization of polymer fiber reinforced composite materials by XPS, SEM, XRD, TEM, and FTIR

#### Research Assistant (1998-2003)

UNIVERSITY OF DELAWARE, Newark, DE

1. Designed and improved procedure for the synthesis of high quality TiO<sub>2</sub> nanoparticles and thin films in MOCVD system.
2. Achieved visible light absorption for wide band gap TiO<sub>2</sub>.
3. Developed a new method for DC magnetron sputtering of high C concentration of metastable Ge<sub>1-x</sub>C<sub>x</sub> epitaxial thin films on Si(100) substrates.
4. Trained students for use of materials synthesis and characterization techniques.

#### Research Assistant (1995-1998)

CHINESE ACADEMY OF SCIENCE, Shenyang, China

1. Designed and synthesized two-phase nanocomposite  $\alpha$ -Fe/Nd<sub>2</sub>Fe<sub>14</sub>B magnetic materials by rapid solidification with annealing procedure.
2. Improved magnetic property of nanosize Nd-Fe-B by addition of metal ions.

Weidong Li's C.V. (Email: [weidong2004@gmail.com](mailto:weidong2004@gmail.com))

**Graduation Practice (1994.8-1994.11)**

Qishuyan Locomotive & Rolling Stock Works, Changzhou, China

1. Conducted structure/composition analysis for train engine parts by EDS and XRD.
2. Performed SEM analysis for mechanical parts failure.

**Summer Intern (1993.6-1993.7)**

Nanjing 2<sup>nd</sup> Chemical Mechanics, Nanjing, China

1. Conducted heat treatment and thermal analysis for metal phase transformation.
2. Materials surface analysis and preparation for welding.

**EDUCATION:**

**Ph.D. in Materials Science 2004.2**

Dept. of Materials Science and Engineering, University of Delaware, Newark, DE

Dissertation: "Metalorganic chemical vapor deposition and characterization of TiO<sub>2</sub> nanoparticles", Advisor: Prof. Ismat Shah

GPA: 3.8/4.0

**M.MSE. 2001.5**

Dept. of Materials Science and Engineering, University of Delaware, Newark, DE

Thesis: "Growth and characterization of Ge<sub>1-x</sub>C<sub>x</sub> epitaxial layers on Si(100) substrates", Advisor: Prof. Ismat Shah

GPA: 3.8/4.0

**M.E. 1998.6**

Institute of Metal Research, Chinese Academy of Sciences, Shenyang, China

Thesis: "The effect of addition element and magnetic annealing on magnetic properties of melt-spun nano-phase Nd-Fe-B alloy", Advisor: Prof. Mingxiu Quan

GPA: 3.5/4.0

**B.E. 1995.7**

Department of Materials Science and Technology,

Nanjing University of Aeronautics and Astronautics, Nanjing, China

GPA: 3.8/4.0

**HONORS AND AWARDS:**

- Member of Sigma Xi the Scientific Research Society, 2005.
- Materials Research Society (MRS) Graduate Research Award, Silver Medal 2003.
- American Vacuum Society (AVS) Graduate Research Award, 2002.
- AVS Russell & Sigurd Varian Fellowship, Finalist 2002.
- AVS Dorothy and Earl S. Hoffman Travel Scholarship, Annually 2000-2003.
- Undergraduate student fellowship, Annually 1992-1994.

**PUBLICATIONS:**

**I: PAPERS**

- 1 H. Lin, C.P. Huang, W. Li, C. Ni, and S.I. Shah, "Size dependency of nanocrystalline TiO<sub>2</sub> on its optical property and photocatalytic reactivity exemplified by 2-Chlorophenol," *Appl. Catal. B*, accepted (2006)
- 2 W. Li, A. Frenkel, J. Woick, C. Ni, and S.I. Shah, "Dopant location identification in Nd<sup>3+</sup>-doped TiO<sub>2</sub> nanoparticles," *Phys. Rev. B* 72, 155315 (2005)
- 3 W. Li, C. Ni, H. Lin, C. P. Huang, and S. I. Shah, "Size dependence of thermal stability of TiO<sub>2</sub> nanoparticles," *J. Appl. Phys.* 96, 6663 (2004).

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- 4 A. Burns, H. Greg, W. Li, J. Hirvonen, J. D. Demaree, and S. I. Shah, "Neodymium-ion dopant effect on the phase transformation of nanostructured TiO<sub>2</sub> synthesized by sol-gel," *Mater. Sci. Eng. B* 111, 150 (2004).
- 5 W. Li, Y. Wang, H. Lin, S.I. Shah, C.P. Huang, D. J. Doren, S. A. Rykov, J.G. Chen, and M.A. Barteau, "Band gap tailoring of Nd<sup>3+</sup>-doped TiO<sub>2</sub> nanoparticles," *Appl. Phys. Lett.* 83, 4143 (2003).
- 6 W. Li, S.I. Shah, C.-P. Huang, O. Jung, and C. Ni, "Metalorganic chemical vapor deposition and characterization of TiO<sub>2</sub> nanoparticles," *Mater. Sci. Eng. B* 96, 247 (2002).
- 7 W. Li, S.I. Shah, M. Sung, and C.-P. Huang, "Structure and size distribution of TiO<sub>2</sub> nanoparticles on stainless steel mesh," *J. Vac. Sci. Technol. B* 20, 2303 (2002).
- 8 W. Li, S.I. Shah, D. Guerin, J.G. Chen, and H. Hwu, "Growth and characterization of epitaxial Ge<sub>1-x</sub>C<sub>x</sub> thin films on (100) Si," *J. Vac. Sci. Technol. A* 19, 2617 (2001).
- 9 S.I. Shah, W. Li, C.-P. Huang, O. Jung, and C. Ni, "Study of Nd<sup>3+</sup>, Pt<sup>4+</sup>, Pd<sup>2+</sup>, and Fe<sup>3+</sup> dopant effect on the photoreactivity of TiO<sub>2</sub> nanoparticles," *PNAS, USA* 99, 6482 (2002).
- 10 W.S. Sun, W. Li, and M.X. Quan, "Remanence enhancement and microstructure in a two-phase Nd<sub>10</sub>Fe<sub>83</sub>B<sub>6</sub>Al<sub>1</sub> nanocrystalline alloy," *Internatl J. of Non-Equilibrium Processing* 10, 297(1998).
- 11 W.D. Li, T.M. Zhao, M.X. Quan, and Z.Q. Hu, "Effect of Al content on glass forming ability, microstructures, and magnetic properties of  $\alpha$ -Fe/Nd<sub>2</sub>Fe<sub>14</sub>B," *Functional Materials (Chinese)*, 29, 232(1998).

## II. CONFERENCE PROCEEDINGS

- 1 S. Chiruvolu, W. Li, M. Ng, K. Du, C. Home, B. McGovern, and R. Mosso et al. "Laser pyrolysis – a platform technology to produce functional nanoscale materials for a range of applications," *Nano Sci. Technol. Inst. (NSTI) 2006 (accepted)*
- 2 A. Burns, W. Li, C. Baker, and S.I. Shah, "Sol-gel synthesis and characterization of neodymium doped nanostructured titania thin films," *Proc. Mater. Res. Soc. Sym.* 703, 193 (2002).

## III: BOOK CHAPTERS

- 1 W. Li and S.I. Shah, "Semiconductor Nanoparticles for Photocatalysis" In *Encyclopedia of Nanoscience and Nanotechnology*, Vol. 9, p. 669-695 (2004). (H. S. Nalwa, Ed., American Scientific Publishers, Stevenson Ranch, CA). ISBN: 1588830659
- 2 S. I. Shah, A. Rumaiz, and W. Li, "Nanostructured Catalysts for Environmental Applications" in *Nanotechnology and the Environment*, (B. Kam, et al. Eds.). American Chemical Society/Oxford University Press, 2005, ISBN: 0841238774

## VI. Patent:

"Highly Crystalline Nanoscale Phosphor Particles and Composite Materials Incorporating the particles", to be filed soon. (Attorney Docket No.: 2950.87US02).

## SELECTED TALKS:

CO-PRESENTER "Laser pyrolysis - a platform technology to produce functional nanoscale materials for a range of applications" NSTI Nanotech, Boston, MA, May 2006.

PRESENTER "Band gap tailoring of Nd doped nanoparticles" MRS symposium, Boston, MA, December 2003.

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PRESENTER "Size dependence of structural, optical, and photocatalytic properties of TiO<sub>2</sub> nanoparticles." 50th AVS International Symposium, Baltimore, MD, November 2003.

PRESENTER "Structural, optical, and photoreactivity of Nd<sup>3+</sup> doped TiO<sub>2</sub> nanoparticles." 49th AVS International Symposium, Denver, CO, November 2002.

PRESENTER "Chemical vapor deposition and characterization of TiO<sub>2</sub> nanoparticles" 48th AVS International Symposium, San Francisco, CA, October 2001.

PRESENTER "Dual magnetron sputtering and characterization of GeC thin films on Si (100) substrates." 47th AVS International Symposium, Boston, MA, October 2000.

CO-PRESENTER "Sol-gel synthesis and characterization of Nd<sup>3+</sup> doped TiO<sub>2</sub> nanostructured thin films." MRS symposium, Boston, MA, November 2001.

CO-PRESENTER "MOCVD synthesis of different dopant doped TiO<sub>2</sub> nanoparticles for photocatalysis." Arthur M. Sackler Colloquium of National Academy of Sciences, Washington DC, May 2001.

#### **SERVICE:**

- **Reviewer/Referee**  
Nanotechnology  
Thin Solid Films  
Journal of Environmental Management  
Industrial & Engineering Chemistry Research  
Central European Journal of Chemistry  
Journal of Physical Chemistry  
Journal of Nanoscience and Nanotechnology  
Journal of Solid State Chemistry
- **Memberships**  
Sigma Xi the Scientific Research Society  
American Vacuum Society (AVS)  
Materials Research Society (MRS)

#### **COMPUTER SKILLS:**

MATLAB, Maple, LabView, AutoCAD, Adobe Photoshop, Macromedia Flash, FrontPage, Dream Weaver, MS Office Tools, C++, Java, Scion Image, XFIT.

#### **REFERENCES (upon request)**





